

VISIT...

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KEEPING A NOTEBOOK

12 KEEPING A NOTEBOOK

A **research notebook** is perhaps one of the most valuable pieces of equipment you can own. With it you can duplicate your work, find out what happened at leisure, and even figure out where you blew it. General guidelines for a notebook are:

1. The notebook must be bound permanently. No loose leaf or even spiral-bound notebooks will do. It should have a sewn binding so that the only way pages can come out is to cut them out. (8 ½ x 11 in. is preferred). **Duplicate carbon notebooks** are available that let you make a carbon copy that is removable and that you can hand in. And if the pages aren't already numbered, you might want to do it yourself.
2. *Use waterproof ink! Never pencil!* Pencil will disappear with time, and so will your grade. Cheap ink will wash away and carry your grades down the drain. Never erase! Just draw *one* line through ~~your errors~~ your errors so that they can still be seen. And never, never, never cut any pages out of the notebook!
3. Leave a few pages at the front for a table of contents.
4. Your notebook is your friend, your confidant. Tell it:
 - a. What you have done. Not what it says to do in the lab book. What you, yourself, have done.
 - b. Any and *all* observations: color changes, temperature rises, explosions..., anything that occurs. Any *reasonable* explanation *why* whatever happened, happened.
5. Skipping pages is *extremely* poor taste. It is **NOT** done!
6. List the **IMPORTANT** chemicals you'll use during each reaction. You should include **USEFUL physical properties**: the name of the compound, molecular formula, molecular weight, melting point, boiling point, density, and so on. You might have entries for the number of moles and notes on handling precautions. Useful information, remember. The *CRC Handbook of Chemistry and Physics*, originally published by the Chemical Rubber Company and better known as the *CRC Handbook*, is one place to get this stuff (see Chapter 3, "Interpreting a Handbook").

Note the qualifier "USEFUL," if you can't use any of the information given, do without it! You look things up *before* the lab so you can tell what's staring back out of the flask at you during the course of the reaction.

Your laboratory experiments can be classified as either of two major types: a technique experiment or a synthesis experiment. Each requires different handling.

A TECHNIQUE EXPERIMENT

In a technique experiment, you get to practice a certain operation *before* you have to do it in the course of a synthesis. Distilling a mixture of two liquids to separate them is a typical technique experiment.

Read the following handwritten notebook pages with some care and attention to the *typeset* notes in the margin. A thousand words are worth a picture or so (Figs. 2-4).

Notebook Notes

1. Use a descriptive title for your experiment. *Distillation*. This implies you've done *all* there is in the *entire* field of distillation. You haven't? Perhaps all you've done is *The Separation of a Liquid Mixture by Distillation*. Hmmmmmm.
2. Writing that first sentence can be difficult. Try stating the obvious.
3. There are no large blank areas in your notebook. Draw sloping lines through them. Going back to enter observations after the experiment is over is *not professional*. Initial and date pages anytime you write anything in your notebook.
4. Note the appropriate changes in verb tense. Before you do the work, you might use the present or future tense when you write about something that *hasn't happened yet*. During the lab, since you are supposed to write what you've actually done just after the time you've actually done it, a simple past tense is sufficient.

A SYNTHESIS EXPERIMENT

In a synthesis experiment, the point of the exercise is to prepare a clean sample of the product you want. All the operations in the lab (e.g., distillation, recrystallization) are just means to this end. The preparation of 1-bromobutane is a classic synthesis and is the basis of the next series of handwritten notebook pages.

Explanatory
titleNumbered
page

6

9/13/86

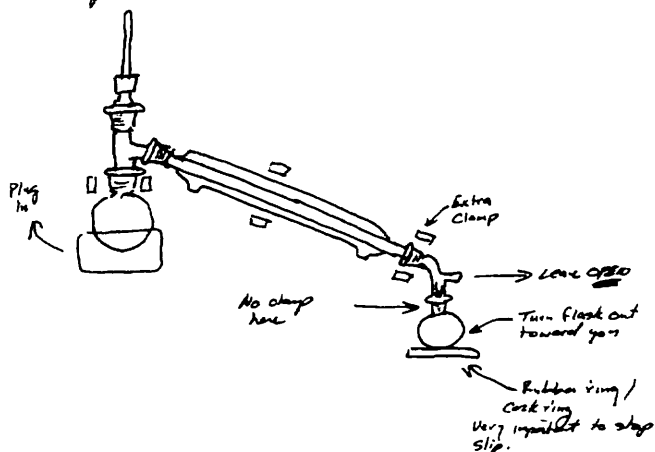
The Separation of a Liquid Mixture by Distillation

This is the
Saturday before
lab.

Distillation is one of the methods of separation and purification of liquids. We will be given an unknown liquid mixture and will have to separate it by distillation.

It's often hard
to start. Hint:
state the obvious.

After we get the unknown, should ^{immediately} dry the liquid over anhydrous magnesium sulfate. The setup is as detailed in the laboratory manual with some changes:



Local procedure
change, probably
from handout.

We will be using Thurnsweil lenses and will not need Variacs. Vacuum adapter clamped at angle, rotated toward me in order to make it easy to change flasks

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Fig. 2 Notebook entry for a technique experiment (1).

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Separation of a Liquid Mixture (cont'd.)

Obtained liquid unknown #20 from instructor & dried it over a slight xs of anhydrous magnesium sulfate. Set up distillation apparatus as described (p. 6). Started with the smallest flask to collect fore-run as suggested by instructor. Filtered unknown into distilling flask with long-stem funnel. Set heat controller to 50. Mixture beginning to boil.

Instant modification.

Liquid condensed on thermometer & temperature reading shot up to 79°C and stabilized at 81°C in a few seconds. Collected $\approx 2\text{ ml}$ as fore-run. Will discard this later. Dropped Thermowall to remove heat to stop distillation and change receiving flask. Started heating again.

Do a bit of work and write a bit of text.

Collected liquid boiling from 81 to 83°C . Changed receiver as above. When new material came over thermometer read $82^{\circ}\text{C}(!)$ for a few minutes (al) then distillation stopped. Temperature began dropping! Turned heat up (70). Mixture starting to boil again and liquid came over @ 123°C . Collected a little of this then changed receiver as above. Shook distilling flask a little & added boiling stone before heating. Had to label flask. So many of them.

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Fig. 3 Notebook entry for a technique experiment (2).

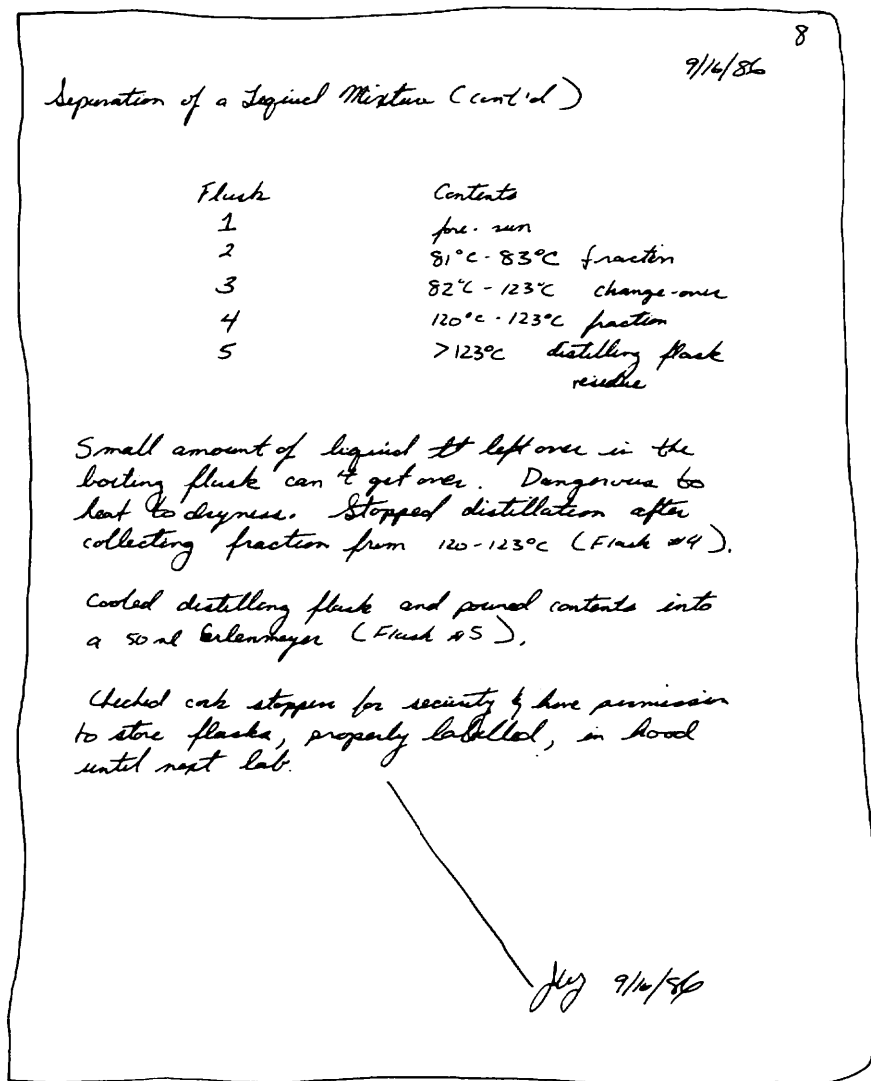


Fig. 4 Notebook entry for a technique experiment (3).

Pay careful attention to the typeset notes in the margins, as well as the handwritten material. Just for fun, go back and see how much was written for the distillation experiment, and note how that is handled in this synthesis (Figs. 5-10).

Once again, if your own instructor wants anything different, do it. The art of notebook keeping has many schools—follow the perspective of your own.

Notebook Notes

1. Use a descriptive title for your experiment. *n*-Butyl Bromide. So what? Did you drink it? Set it on fire? What?! *The Synthesis of 1-Bromobutane from 1-Butanol*—now *that's* a title.
2. Do you see a section for unimportant side reactions? No. Then don't include any.
3. In this experiment, we use a 10% aqueous sodium hydroxide solution as a wash (see Chapter 15, "Extraction and Washing") and anhydrous calcium chloride as a drying agent (see Chapter 10, "Drying Agents"). These are not listed in the Table of Physical Constants. They are neither reactants nor products. Every year, however, somebody always lists the physical properties of *solid* sodium hydroxide, calcium chloride drying agent, and a bunch of other reagents that have nothing to do with the main synthetic reaction. I'm especially puzzled by the listing of solid sodium hydroxide in place of the 10% solution.
4. **Theoretical yield** (not yeild) calculations always seem to be beyond the ken of a lot of you, even though these are exercises right out of the freshman year chemistry course. Yes, we do expect you to remember some things from courses past, the least of which is where to look this up. I've put a sample calculation in the notebook (Fig. 6), that gets the mass (g) of the desired product (1-bromobutane) from the volume (ml) of one reactant (1-butanol). Why from the 1-butanol and not from the sulfuric acid or sodium bromide? It's the 1-butanol we are trying to convert to the bromide, and we use a **molar excess** (often abbreviated XS) of everything else. The 1-butanol is, then, the **limiting reagent**: the reagent present in the smallest molar ratio. Note the use of the density to get from volume to mass (ml to g), molecular weight to go from mass to number of moles (g to mol), the stoichiometric ratio (here 1:1) to get moles of product from moles of limiting reagent, and, finally, reapplication of molecular weight to get the mass (g) of the product.

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Synthesis of 1-Bromobutane

We will be preparing 1-bromobutane as follows: Main reaction.

(1) $\text{CH}_3\text{CH}_2\text{CH}_2\text{CH}_2\text{OH} + \text{NaBr} \xrightarrow{\text{H}_2\text{SO}_4} \text{CH}_3\text{CH}_2\text{CH}_2\text{CH}_2\text{Br} + \text{H}_2\text{O} + \text{Na}_2\text{SO}_4$

Side reactions:

(2) $\text{CH}_3\text{CH}_2\text{CH}_2\text{CH}_2\text{OH} \xrightarrow{\text{H}_2\text{SO}_4} (\text{CH}_3\text{CH}_2\text{CH}_2\text{CH}_2)_2\text{O}$

(3) $\text{CH}_3\text{CH}_2\text{CH}_2\text{CH}_2\text{OH} \xrightarrow{\text{H}_2\text{SO}_4} \text{CH}_3\text{CH}_2\text{CH}=\text{CH}_2 + \text{H}_2\text{O}$

(4) $\text{CH}_3\text{CH}_2\text{CH}_2\text{CH}_2\text{OH} \xrightarrow{\text{H}_2\text{SO}_4} \text{CH}_3\text{CH}=\text{CH}-\text{CH}_3 + \text{H}_2\text{O}$ Important side reactions.

Table of Physical Constants: Physical constants you'll need during your experiment.

NAME	FORMULA	M.W.	Dens.	M.P. °C	B.P. °C	water	ether	conc. H ₂ SO ₄	other
1-Butanol	$\text{CH}_3\text{CH}_2\text{CH}_2\text{CH}_2\text{OH}$	74.12	0.8098	-89.8	117.5	S	∞		s. alc.
Sulfuric Acid	H_2SO_4 98% 100%	98.08 102.9	1.825 1.825			with heat S			
Sodium Bromide	NaBr	102.9							
1-Bromobutane	$\text{CH}_3\text{CH}_2\text{CH}_2\text{CH}_2\text{Br}$	137.03	1.2764	-112.3	101.3	i	S	i	s. alc.
n-dibutyl ether	$(\text{C}_4\text{H}_9)_2\text{O}$	130.23			142	S. sol	∞		∞ alc.
trans-2-butene	$\text{CH}_3\text{CH}=\text{CHCH}_3$	56.10			2.5	i		reacts	vs. alc.
cis-2-butene	$\text{CH}_3\text{CH}=\text{CHCH}_3$	56.10			1	i		reacts	
1-butene	$\text{CH}_2=\text{CHCH}_2\text{CH}_3$	56.10			-5	i	v.s.		vs. alc.

Fig. 5 Notebook entry for a synthesis experiment (1).

Calculations from
freshman chemistry.

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Synthesis of 1-Bromobutane (cont'd.)

Theoretical yield of 1-bromobutane based on 17 ml 1-butanol. Mass of liquid
from the density.

$$17 \text{ ml 1-butanol} \times \frac{0.8098 \text{ g 1-butanol}}{1 \text{ ml 1-butanol}} = 13.77 \text{ g 1-butanol}$$

Moles of
starting material.

$$13.77 \text{ g 1-butanol} \times \frac{1 \text{ mole 1-butanol}}{74.12 \text{ g 1-butanol}} = 0.1857 \text{ moles 1-butanol}$$

$$0.1857 \text{ moles 1-butanol} \times \frac{1 \text{ mole 1-bromobutane}}{1 \text{ mole 1-butanol}} = 0.1857 \text{ moles}$$

1-bromobutane
Moles of
product

$$0.1857 \text{ moles 1-bromobutane} \times \frac{137.03 \text{ g 1-bromobutane}}{1 \text{ mole 1-bromobutane}} = 25.44 \text{ g (calculated).}$$

Grams of
product (calculated).
This is
theoretical yield.

Apparatus: Reflex setup as in Zaitsev with gas
(HBr) trap at top of reflux condenser:

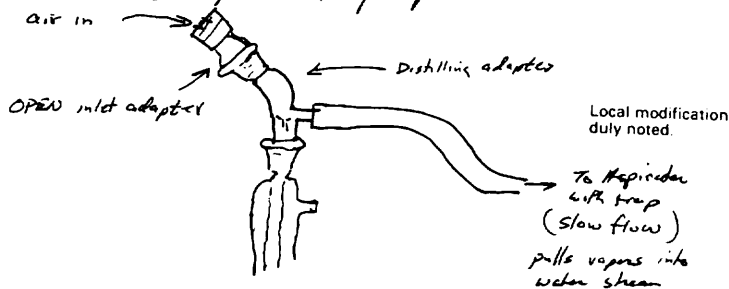


Fig. 6 Notebook entry for a synthesis experiment (2).

Synthesis of 1-Bromobutane (cont'd)

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Outline of Procedure.

1. Place 24.0 g NaBr, 25 ml water, and 17 ml 1-butanol in a 250 ml R.B. flask and cool in ice-water bath to $< 10^{\circ}\text{C}$.
2. SLOWLY add 20 ml. conc. H_2SO_4 with swirling.
3. Reflux this mixture over a flame for 30 min.
4. Let mixture cool. Distill mixture - receiver cooled in ice-water bath. Distill until distillate is NOT cloudy.
5. Collect a few drops of clear distillate in a test-tube. Add water and shake tube. If two layers form, continue distilling another 5-10 min. and repeat this test. If two layers do not form, distill for another 5-10 minutes and quit.
6. Wash distillate with 25 ml H_2O .
7. Wash distillate with 15 ml cold conc. H_2SO_4 .
8. Wash distillate with 15 ml 10% sodium hydroxide solution.
9. Dry with anhydrous MgSO_4 & filter (gravity) into small, dry R.B. flask.
10. Distill dried product using dried distillation apparatus.

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Fig. 7 Notebook entry for a synthesis experiment (3).

Synthesis of 1-Bromobutane (cont'd.)

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10/7/86

Placed 24.0 g NaBr, 25 ml water and 17 ml 1-butanol in a 250 ml R.B. flask & let cool in an ice-bath. When liquid reached 5°C, added H₂SO₄ with swirling. The mixture warmed up and turned a yellow color.

Set up for reflux with gas trap as on p. 27. Mixture darkened as reflux continued.

Next step performed while reflux continues.

Placed 15 ml H₂SO₄ in separatory funnel clamped to cool in ice-water bath for later.

After refluxing 30 min, let reaction mixture cool to room temp, then put in ice-water bath. There are two distinct layers in the flask, both an orange color. Color may be due to free bromine. One of the two layers is product.

Only a phrase recalls the distillation.

Distilled the mixture, and collected everything that came over up to 100°C. Initially, cloudy, white liquid came over (water + organic product?) then clear liquid. Stopped heating, quickly flask, removed receiver and replaced it with test tube in beaker with ice & water. Noted to distill over a few drops of liquid. Added a bit more than an equal amount of water - shook tube. NO LAYERS FORMED! Replaced receiving flask & distilled for 5 min. more.

Poured distillate into a 125 ml separatory funnel & added 25 ml water. Water went into upper layer - upper layer is aqueous; lower is organic product. Save Lower Layer.

Note the recorded observations.

Fig. 8 Notebook entry for a synthesis experiment (4).

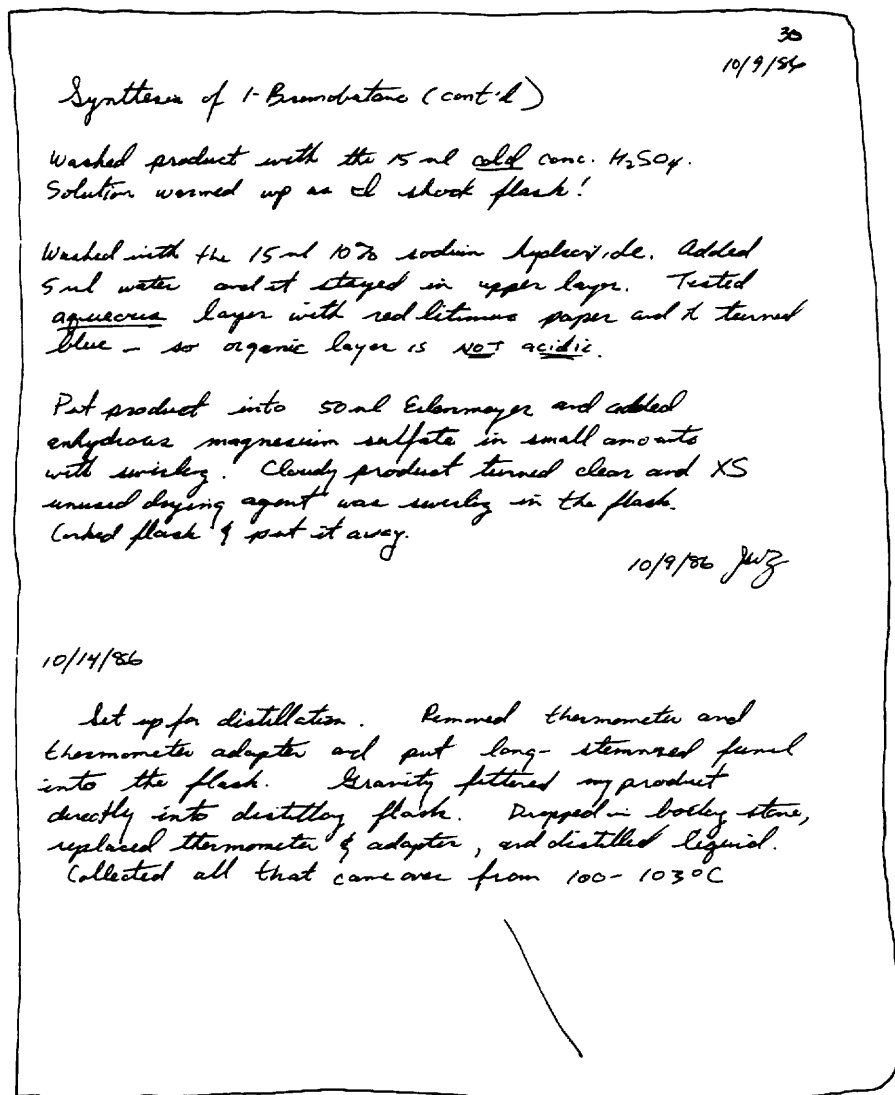


Fig. 9 Notebook entry for a synthesis experiment (5).

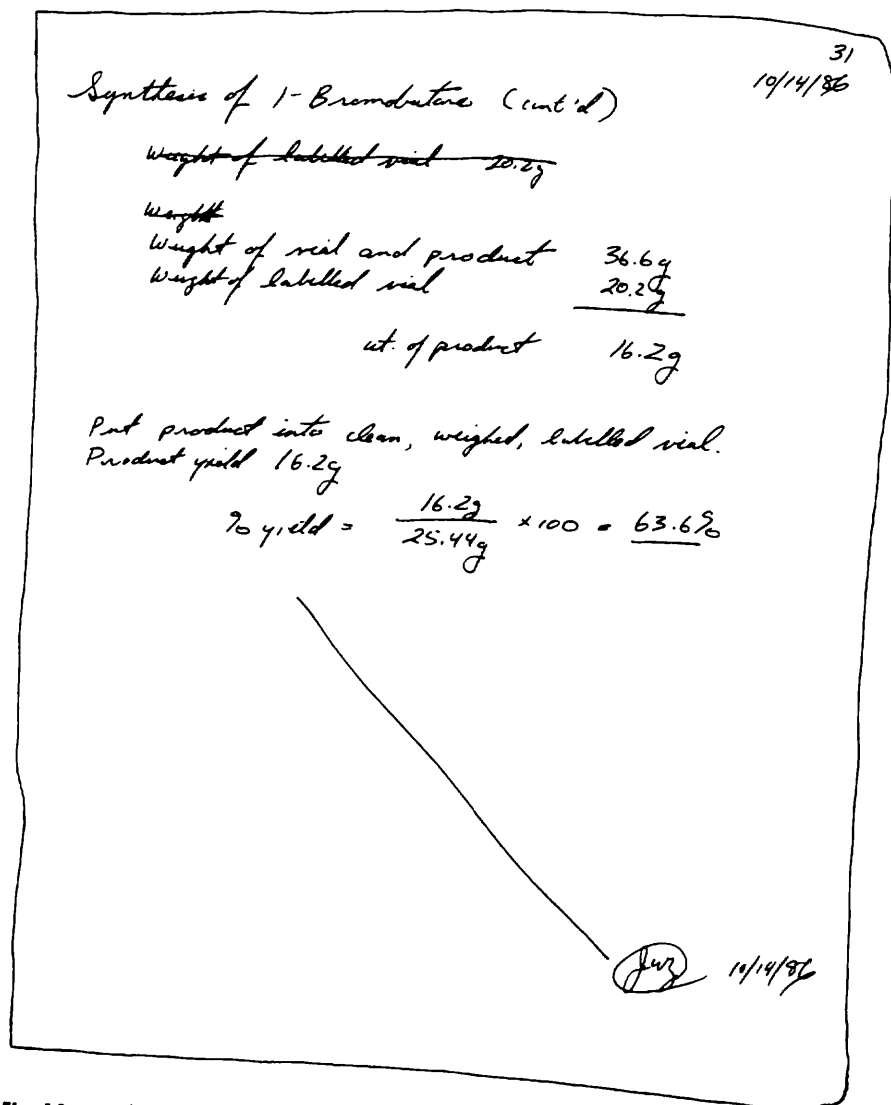


Fig. 10 Notebook entry for a synthesis experiment (6).

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Note that this mass is *calculated*. It is NOT anything we've actually produced. In theory, we get this much. That is theoretical yield.

5. I'm a firm believer in the use of units, factor-label method, dimensional analysis, whatever you call it. I know I've screwed up if my units are (g 1-butanol)²/mole 1-butanol.
6. Remember the huge writeup on the *Separation of a Liquid Mixture by Distillation*, drawings of apparatus and all? Well, the line "the mixture was purified by distillation" (Fig. 9) is all you write for the distillation during this synthesis.
7. At the end of the synthesis, you calculate the **percent yield**. Just divide the amount you *actually prepared* by the amount you calculated you'd get, and multiply this fraction by 100. For this synthesis, I *calculated* a yield of 25.44 g of product. For this reaction on the bench, I *actually* obtained 16.2 g of product. So:

$$(16.2 \text{ g} / 25.44 \text{ g})(100) = \mathbf{63.6\% \text{ yield}}$$

Note that's not 63.624715321%. **Significant figures**, please. The product is weighed to one part in ten ([+ | 0.1) and calculated to one part in one hundred ([+ | 0.01). If the product weight can vary by [+ | 0.1 g, what's the use of all those figures?

THE ACID TEST

After all this, you're still not sure what to write in your notebook? Try these simple tests at home.

1. Before lab. "Can I carry out this experiment without any lab manual?"
2. After lab. (I mean *immediately* after; none of this "I'll write my observations down later" garbage.) Ask yourself: "If this were someone else's notebook, could I duplicate the results exactly?"

If you can truthfully answer yes to these two questions, you're doing very well indeed.